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Indian Standard

**METHODS FOR IDENTIFICATION OF
THE APPLICATION CLASSES OF DYES ON
TEXTILE MATERIALS**

PART I COTTON AND OTHER CELLULOSIC FIBRES

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

*Indian Standard*METHODS FOR IDENTIFICATION OF
THE APPLICATION CLASSES OF DYES ON
TEXTILE MATERIALS

PART I COTTON AND OTHER CELLULOSIC FIBRES

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Indian Standard

METHODS FOR IDENTIFICATION OF THE APPLICATION CLASSES OF DYES ON TEXTILE MATERIALS

PART I COTTON AND OTHER CELLULOSIC FIBRES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 December 1967, after the draft finalized by the Dyestuffs Sectional Committee had been approved by the Textile Division Council.

0.2 The methods described in this standard are simple and are meant for a rapid identification of the application classes of dyestuffs. Though the methods prescribed here do not involve the use of microscope, its value in the identification cannot be over-emphasized; when one is available it should always be used for the identification.

0.3 Successful identification of the application classes of dyestuff depends on experience and familiarity of the dyestuffs. In general the identification of the application classes of dyestuffs is not dependant on any single test.

0.4 Considerable assistance has been derived from the following.

Identification of dyestuffs on textiles. *Textile industries journal*. 122, 1; 1958; 101-104.

ELLIS CLAVTON. Identification of dyes on textile fibres. 1963. Ed 2. The Society of Dyers and Colourists, Bradford, U.K.

1. SCOPE

1.1 This standard prescribes methods for identification of application classes of dyes on cotton and other cellulosic fibres.

1.1.1 The methods are applicable to types of dyes normally used for dyeing and printing cotton and other cellulosic fibres.

2. PREPARATION OF TEST SPECIMEN

2.1 If the sample under test is fibre or yarn, take a tuft of fibre or yarn of about 3 cm in length.

2.2 If the sample under test is fabric, take a 3×3 cm test piece.

NOTE 1 — In case of multi-coloured woven fabrics, the different coloured yarn should be identified separately.

NOTE 2 — In case of printed fabrics the sample should be taken from the printed portion of the sample.

2.3 In the case of finished textiles, the sample should be treated twice with 1 percent hydrochloric acid at boil for 5 minutes.

3. REAGENTS

3.0 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the test results.

3.1 Ammonium Hydroxide — (a) dilute (1 percent w/v), and (b) concentrated [25 percent (w/v) or sp gr 0.90].

3.2 Sodium Hydroxide Solution — (a) 5 percent (w/v), (b) 10 percent (w/v), and (c) 44 percent (w/v) (or 70°TW).

3.3 Sodium Carbonate — (a) 5 percent solution (w/v), and (b) solid.

3.4 Ammonium Chloride Solution — 5 percent (w/v).

3.5 Vat Dye Developer Solution — prepared by dissolving 8 g of ammonium chloride and 2 g of ammonium persulphate in 100 ml of water.

3.6 Hydrogen Peroxide — 3 percent (w/v) (10 volumes).

3.7 Sodium Sulphoxylate Formaldehyde — Glycol Solution — prepared by dissolving 20 g of sodium sulphoxylate formaldehyde in 75 ml of warm water and diluted with cold water. To this is added 50 g of mono- or diethylene glycol.

NOTE — Sodium sulphoxylate formaldehyde is commercially available as ' Formosul ' ' Rongalite ', etc.

3.8 Ethylenediamine — sp gr 0.97, b.p. 117°C.

3.9 Acetic Acid — (a) 10 percent (w/v), (b) 20 percent (w/v), and (c) glacial.

3.10 Hydrochloric Acid — (a) 1 percent (w/v), and (b) 10 percent (w/v).

3.11 Sodium Hypochlorite Solution — 2 to 3 g/l of available chlorine.

3.12 Sodium Sulphide — (a) 5 percent solution (w/v), and (b) solid.

3.13 Tannin Reagent — prepared by dissolving 10 g of tannic acid and 10 g of anhydrous sodium acetate in 200 ml of water.

*Specification for water, distilled quality (revised).

3.14 Lead Acetate Solution — 5 percent (*w/v*).

3.15 Stannous Chloride Solution — prepared by dissolving 100 g of stannous chloride in 100 ml of concentrated hydrochloric acid at boil.

3.16 Rectified Spirit — *see* IS : 323-1959*.

3.17 Ferric Chloride Solution — 1 percent (*w/v*).

3.18 Glucose — solid.

3.19 Sulphuric Acid — (a) 5 percent (*w/v*), (b) 12 N, and (c) concentrated (sp gr 1.84).

3.20 Sodium Nitrate — solid.

3.21 Nitric Acid — concentrated (sp gr 1.42).

3.22 Carbazol Solution — 1 percent solution in concentrated sulphuric acid.

3.23 Chromotropic Acid — 5 percent solution in distilled water.

3.24 Pyridine — b.p. 112° to 114°C.

3.25 Sodium Hydrosulphite — solid (*see* IS : 1919-1961†).

3.26 Dimethylformamide — (a) 50 percent (*w/v*), and (b) concentrated b.p. 152° to 154°C.

3.27 Phenol

3.28 Monoethyl Ether of Ethylene Glycol — b.p. 132° to 136°C.

3.29 Mordanted Cotton — mordanted with tannic acid or any other mordant suitable for basic dyes.

3.30 Bleached Cotton

3.31 Undyed Wool

3.32 Sodium Chloride — solid.

3.33 Methyl Pyrrolidone — b.p. 202°C.

4. PROCEDURE

4.1 For preliminary identification of dyes on fibres, follow the procedure given in Appendix A.

4.2 Confirm, wherever necessary, the class of dyes by the tests prescribed in Appendix B.

NOTE — While identifying the dyes used for dyeing pale shades, it is advisable to use large specimen and larger quantities of reagents and to concentrate the extract before making the test.

4.3 For the dyes which are not easily identified by the procedure given in Appendices A and B, follow the procedure given in Appendix C.

*Specification for rectified spirit (*revised*).

†Specification for sodium hydrosulphite, technical.

APPENDIX A

(Clause 4.1)

PRELIMINARY TESTS FOR IDENTIFICATION OF DYES ON COTTON AND OTHER CELLULOSIC FIBRES

The test specimen is successively treated with (a) 50-percent dimethylformamide, (b) concentrated dimethylformamide, and (c) a mixture of glacial acetic acid and rectified spirit (1 : 1, v/v) at boil for 3 to 4 minutes with intermediate washing with water and squeezing.

If the dyes of Group I are absent, take a fresh test specimen and boil it for 1 to 2 minutes in 1 percent ammonium hydroxide solution.

If the test specimen does not bleed (or bleeds slightly), take a fresh test specimen and add 1 ml of glacial acetic acid and warm. Add 3 to 5 ml of water and boil. Remove the specimen and add 25 mg of mordanted cotton and boil for 2 minutes.

If direct, reactive (ingrain except azoics) acid and basic dyes are absent, take a fresh test specimen, add 10 to 15 ml of 1-percent hydrochloric acid. Boil for 1 minute. Discard the acid solution and repeat this once or twice.

If the dyes of Group II are absent, a fresh test specimen is boiled for 1 to 2 minutes in solution containing 10 to 30 mg of sodium hydrosulphite in 5 to 10 ml of water to which 4 to 6 drops of 44 percent sodium hydroxide solution are added.

Group III dyes decolourize or change shade radically. The colour of the test specimen is restored on exposure to air or with vat-dye developer.

All Group IV dyes are destroyed and on re-oxidation do not restore to the original colour.

<p>No stripping or partial stripping of dye. <i>Reactive Dyes and Ingrain Dyes (Except Azoics)</i> (Some of the basic dyes are not stripped)</p>	<p>The specimen bleeds off the dye. The solution is distinctly coloured. Remove the test specimen and add a few pieces of white bleached cotton, 25 mg of sodium chloride and boil for 2 minutes. Cool and rinse the added bleached cotton. The white cotton is dyed to approximately original shade. <i>Direct Dyes</i></p> <p>The specimen bleeds off the colour and the white cotton is left undyed or slightly stained, neutralize the solution with acetic acid and add 1 ml of 10 percent acetic acid. Add undyed wool pieces and boil for 1 minute. Cool and rinse the added wool pieces. The wool is dyed. <i>Acid Dyes (If Direct and Basic Dyes Absent)</i></p>	<p>Mordanted cotton is dyed. <i>Basic Dyes</i></p>	<p>Test the specimen (treated with 1 percent hydrochloric acid) for the presence of direct dyes if the specimen responds to direct dyes test. <i>Direct Dyes After-treated With Resin</i></p> <p>Take a fresh test specimen, add 2 to 3 ml of water, 1 to 2 ml of 5 percent sodium carbonate solution, 500 mg of solid sodium sulphate and boil for 2 minutes. Remove the specimen. Add 25 mg of sodium chloride and a few pieces of white bleached cotton. Boil for 2 minutes; the white cotton is dyed. Place the test specimen and white cotton on filter paper and reoxidize the dye and dyeings on white cotton. White cotton is redyed to original shade (except strength) and the test specimen restores the colour. <i>Sulphur Dyes</i></p> <p>Cotton is not redyed from sodium carbonate-sodium sulphide solution. Take a fresh sample in an evaporating dish, pour 2 to 3 ml of concentrated sulphuric acid. Shake just enough to extract the dye. Pour the extract into the test tube, add 25 ml of water and filter. Wash the filter paper with water. Spot the filter paper with 10 percent sodium hydroxide solution. Spot turns red violet. <i>Oxidation Blacks (Aniline Black)</i></p> <p>If sulphur and oxidation blacks are absent, take a fresh test specimen; boil it with sodium sulphoxylate formaldehyde-glycol solution containing few drops of sodium hydroxide solution (44 percent). Test specimen is decolourized or altered in shade. The solution is yellow, bluish red, etc. Original colour restored by vat-dye developer. <i>Vat Dyes</i></p>	<p>Take a fresh test specimen (about 6 g) and ash it in a porcelain crucible. Add 200 mg of flux (equal parts of sodium carbonate and sodium nitrate and fuse. Fused mass is orange yellow when hot and permanent greenish yellow when cold. <i>Direct Dyes After-treated With Chromium Salts</i></p> <p>If chromium is absent, ash the test specimen as above and dissolve the ash in few drops of concentrated nitric acid. Add 2 ml of water, boil and cool. Add 2 ml of concentrated ammonium hydroxide. Blue colour indicates: <i>Direct Dyes After-treated with Copper Salts</i></p> <p>If chromium or copper salts treated direct dyes are absent, take a fresh test specimen, treat it with 5 percent boiling sulphuric acid. Cool and add dropwise 1 percent corbazol solution. Blue precipitate indicates presence of formaldehyde. <i>Direct Dyes After-treated with Formaldehyde</i></p> <p>Specimen bleeds and continues to do so in subsequent treatment. <i>Azoic Dyes</i></p> <p>Specimen does not bleed or bleeds slightly; bleeding decreases or in most cases terminate. <i>Diazotized and Developed Dyes</i></p>
Group I	Group II		Group III	Group IV
				Group V

The dyes of Group V usually do not respond to the reactions of the first four groups (if they do the reactions are slower and often incomplete).
Vat and Azoic Pigments

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APPENDIX B

(Clause 4.2, and Appendix A)

**CONFIRMATORY TESTS FOR IDENTIFICATION
OF DYES ON COTTON AND OTHER
CELLULOSIC FIBRES**

B-1. DIRECT DYES

B-1.1 Take a test specimen. Boil it for half a minute in 5 percent sodium hydroxide solution (*see Note under B-1.2*). Add little mercerized cotton to the solution. Dye the mercerized cotton for 10 minutes. The dye on the mercerized cotton is not stripped by 1 percent ammonium hydroxide solution.

B-1.2 Take a test specimen. Add to it little cold ethylenediamine and shake for some time. Dilute the coloured extract with water. Introduce a few pieces of white cotton and heat to 80°C. Add little sodium chloride and allow it to cool. The white cotton is stained evenly and the stain is not removed by boiling with 1 percent ammonium hydroxide solution.

NOTE — Certain pale blue dyeings are not appreciably affected by boiling 5 percent sodium hydroxide solution. However, they are stripped to white in about 15 minutes by cold ethylenediamine. Dyes of this type belong to 'dioxazine' series.

B-1.3 Direct Dyes After-treated with Formaldehyde — Take a test specimen. Extract it with 12 N sulphuric acid for 5 minutes. Add 1 to 2 ml of concentrated sulphuric acid and 4 to 5 drops of chromotropic acid. Reddish violet colour is obtained.

B-2. BASIC DYES

B-2.1 Take a test specimen. Add to it 1 ml of 5 percent sodium hydroxide solution and boil for a few seconds. To this add 4 ml of 5 percent ammonium chloride solution and boil again.

B-2.1.1 Take small amount of extract obtained as in **B-2.1**. Add to it a few pieces of undyed wool and allow the solution to cool. Most of the dye is taken up by the wool.

B-2.1.2 Take small amount of extract obtained as in **B-2.1**. Acidify the solution with 10-percent acetic acid and add tannin reagent. A coloured precipitate is obtained.

B-2.1.3 Take small amount of extract obtained as in **B-2.1**. Acidify the solution with 10-percent acetic acid. Add to it 1 percent solution of ferric chloride. A black precipitate is obtained.

NOTE — The tannin mordanted dyeings would give the above test.

B-3. SULPHUR DYES

B-3.1 Take a test specimen. Boil with stannous chloride solution in a test tube, the mouth of which is covered with filter paper moistened with lead acetate solution. Brown stains on filter paper will indicate sulphur dyes.

NOTE 1 — Deep brown stains should be regarded as positive test.

NOTE 2 — Prolonged boiling may discolour the brown stains due to action of hydrochloric acid.

B-3.2 Take a test specimen. Boil it with ethylenediamine. The dye is readily stripped.

NOTE — Indocarbon black dyeings give reddish solution which revert to original shade on dilution with water whereas sulphur black dyeings give greenish solution.

B-3.3 Take a test specimen. Treat it with sodium hypochlorite solution. The specimen is bleached to white or buff colour.

NOTE — Indocarbon blacks are not affected by this test.

B-4. VAT DYES

B-4.1 Take a test specimen. Boil it with 5 to 10 ml of sodium sulfoxylate formaldehyde-glycol solution containing little 44 percent sodium hydroxide solution. A distinct change in colour is observed. Take out the test specimen and wash with fresh water. The original colour is restored, if not, treat the test specimen with vat dye developer or hydrogen peroxide.

B-4.2 Take a test specimen. Treat it with little ethylenediamine and glucose. The colour is more or less completely removed when treated near the boiling point.

B-5. AZOIC DYES

B-5.1 Take a test specimen, add sufficient amount of ethylenediamine, boil for few minutes a considerable amount of dye is extracted. Divide the extract in two parts and proceed as below.

B-5.1.1 To one part of the extract, add a little sodium hydrosulphite. Warm if necessary, permanent decolourization occurs.

B-5.1.2 Dilute the other part of extract with water and boil. The liquid becomes turbid and the flakes of coloured pigment settle down on standing.

B-5.2 Take a test specimen. Add to it little sodium sulfoxylate formaldehyde-glycol solution containing 44 percent sodium hydroxide solution and boil vigorously. Most of the dyeings are reduced to colourless or yellow compounds.

NOTE — If no signs of reduction appear after boiling for 1 or 2 minutes, treat the test specimen in boiling 5 percent sodium hydroxide solution, to which little sodium hydrosulphite is added. All azoic dyeings including the yellow ones are reduced to pale yellow or white by this method. Some of the alkaline reduction products exhibit fluorescence when exposed to ultra-violet rays. This phenomenon is of value in confirming the presence of certain pale azoic dyeings and prints.

B-5.3 Take a test specimen. Dip it in liquid phenol. Take out the specimen and squeeze it lightly. Lay the test specimen between two filter papers, and press it either with hot iron or on steam pipe. The filter paper is stained.

B-5.4 Take a test specimen. Add to it a solution of equal volumes of 10-percent sodium hydroxide and monoethyl ether of ethylene glycol, and little sodium hydrosulphite. The azoic dyeings are reduced to colourless or yellow compound on boiling.

NOTE — The above test is carried out especially when the dyeings are on the viscose rayon.

B-6. PIGMENTS

B-6.1 Vat Pigments — Take a test specimen and treat it with methyl pyrrolidone which will plasticize the resin binder. Carry out tests for the presence of vat dyes.

B-6.2 Azoic Pigments — Take a test specimen weighing about 200 mg and treat it with 1 ml of methyl pyrrolidone for 30 seconds and cool. Add 5 percent sodium hydroxide solution and 25 to 50 mg of sodium hydrosulphite. Boil till the sample is white, light yellow or orange. Filter the solution and add 25 mg of sodium chloride and a few pieces of cotton. Boil for 1 minute and cool. Take out the white cotton and dry. Naphthol AS pigments would be yellow while β -naphthol pigments would darken and turn brown.

B-7. OXIDATION BLACK (ANILINE BLACKS)

B-7.1 Take a test specimen and digest it with concentrated sulphuric acid in cold. Dilute the solution with water, a green colour is obtained.

B-7.2 Take a test specimen. Treat it for 1 minute with sodium hypochlorite solution, the specimen turns brown.

B-7.3 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron or copper; a positive test for the presence of iron or copper is obtained.

B-8. INGRAIN DYES (EXCEPT AZOICS)

B-8.1 Phthalogen Green

B-8.1.1 Take a test specimen. Add 1 ml of methyl pyrrolidone. Heat to boil and cool to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen reduces to dark violet. Remove the test specimen and put it in 20 percent acetic acid solution. The colour remains violet.

B-8.2 Phthalogen Blue

B-8.2.1 Take a test specimen. Add 1 ml of methyl pyrrolidone. Heat to boil and cool to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen does not reduce.

B-8.2.2 Take a test specimen and spot it with concentrated nitric acid. The colour changes to violet.

B-8.2.3 Take a test specimen and spot it with concentrated sulphuric acid. The colour changes to bright green.

B-8.3 Alcian Blue

B-8.3.1 Take a test specimen. Add 1 ml of methyl pyrrolidone. Heat to boil and cool it to 70°C. Add 1 to 2 ml of 10 percent sodium hydroxide solution, 20 to 40 mg of sodium hydrosulphite and heat to boil. The colour of the test specimen changes to violet. Remove the test specimen and put it in 20 percent acetic acid solution. The colour of the test specimen changes to green.

B-8.3.2 Take a test specimen and spot it with concentrated nitric acid. The colour of the specimen changes to violet.

B-8.3.3 Take a test specimen and spot it with concentrated sulphuric acid. The colour of the test specimen changes to green.

A P P E N D I X C

(Clause 4.3)

METHOD OF TEST FOR MINERAL KHAKI, VEGETABLE KHAKI, CATECHU, ETC

C-1. MINERAL KHAKI

C-1.1 Take a test specimen. Boil it for 5 minutes with 10 percent hydrochloric acid. Observe the test specimen. The colour of the test specimen is completely stripped off and the solution is green.

C-1.2 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron and chromium. The positive test for iron and chromium indicates the presence of mineral khaki.

C-2. VEGETABLE KHAKI

C-2.1 Take a test specimen. Boil it for 5 minutes with 10 percent hydrochloric acid. Observe the test specimen. The colour of the test specimen is not completely stripped off and the solution is yellow.

C-2.2 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of iron and chromium. The positive test for iron and chromium indicates the presence of vegetable khaki.

C-2.3 Take a test specimen and treat it with 5 percent sodium hydroxide solution at boil for 1 to 2 minutes; the specimen as well as the solution turns brownish.

C-3. CATECHU

C-3.1 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of copper and chromium. The positive test for copper and chromium indicates the presence of catechu.

C-3.2 Take a test specimen. Put a drop of concentrated nitric acid on it, an orange colour is obtained.

C-4. CHROME YELLOW AND CHROME ORANGE

C-4.1 Take a test specimen (about 5 g) and ash it. Test the ash for the presence of lead. The positive test of lead indicates the presence of chrome yellow or chrome orange.

NOTE — Chrome yellow dyeing on treatment with 5 percent sodium hydroxide solution becomes orange and chrome orange dyeing on treatment with 10 percent hydrochloric acid becomes yellow.

C-5. PRUSSIAN BLUE

C-5.1 Take a test specimen (about 5 g) and ash it. Test the ash in the presence of iron. The positive test for iron indicates presence of prussian blue.

C-5.2 Take a test specimen and treat it with hot 5 percent sodium hydroxide solution. The specimen turns brown.

IS : 4472 (Part I) - 1967

(Continued from page 1)

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